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The reclamation of useful products from solid waste is one method of reducing the disposal problem of solid wastes. There are many research projects concerned with recycling solid wastes into fertilizers and food products. This report describes and evaluates one method for measuring phosphorus in the form of phosphates that are present in solid wastes. The vanadomolybdophosphoric acid method was selected to be investigated as a method for solid waste analyses because of its high range, simplicity, and freedom from interferences. The chemistry which makes this method useful for determining phosphates is believed to involve the subsitution of oxyvanadium and oxymolybdenum radicals for the O of P04 to give a heteropoly compound that is chromogenic. The intensity of yellow color produced is proportional to the concentration of phosphates present; thus the phosphates can be determined colormetrically by measuring the intensity of the yellow color produced when P04 comes in contact with vanadate and molybdate in an acid solution.

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DETERMINATION OF THE PHOSPHATE IN SOLID WASTE USING THE VANADOMOLYBDOPHOSPHORIC ACID METHOD

A Solid Waste Research
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written by
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TABLE OF CONTENTS

	Page
INTRODUCTION	1
EXPERIMENTAL	2
Equipment	2
Reagents	2
Procedure	3
DISCUSSION	5
TABLES	7
REFERENCE	10
ACKNOWLEDGEMENT	10

DETERMINATION OF THE PHOSPHATE IN SOLID WASTE USING THE VANADOMOLYBDOPHOSPHORIC ACID METHOD

William H. Kaylor*

INTRODUCTION

The reclamation of useful products from solid waste is one method of reducing the disposal problem of solid wastes. There are many research projects concerned with recycling solid wastes into fertilizers and food products. In the production of fertilizers and food products from solid waste, the phosphorous concentration is of importance because phosphorus is a nutrient. It is also important to know the phosphate concentration in solid waste leachates, since these leachates may eventually drain into natural water supplies and cause pollution if standard concentration limits are exceeded. This report describes and evaluates one method for measuring phosphorus in the form of phosphates that are present in solid wastes.

The vanadomolybdophosphoric acid method[1] was selected to be investigated as a method for solid waste analyses because of its high range, simplicity, and freedom from interferences. The chemistry which makes this method useful for determining phosphates is believed to involve the substitution of oxyvanadium and oxymolybdenum radicals for the O of PO_4 to give a heteropoly compound that is chromogenic.

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The intensity of yellow color produced is proportional to the concentration of phosphates present; thus the phosphates can be determined colormetrically by measuring the intensity of the yellow color produced when PO_4 comes in contact with vanadate and molybdate in an acid solution.

EXPERIMENTAL

Vanadomolydophosphoric Acid Method for Phosphates

1. Equipment
   a. Erlenmeyer flasks - 125 ml
   b. Kjeldahl flasks - 800 ml
   c. Volumetric pipettes - 5, 10 and 20 ml
   d. Filtering funnels
   e. Filter paper - only specification is phosphate free
   f. Color comparison tubes or volumetric flasks - 50 ml
   g. Spectrophotometer - wave length setting 400 mu
2. Reagents
   a. Ammonium vanadate - molybdate solution: Dissolve 2.35 g ammonium metavanadate in 300 ml of boiling distilled water; cool, then add 400 ml concentrated HNO_3 and cool. Dissolve 40 g ammonium molybdate in 400 ml hot distilled water and cool. The molybdate solution is then added to the vanadate solution and diluted to 2 liters with distilled water.

b. Sulfuric acid solution: Add 310 ml concentrated H_2SO_4 cautiously to 500 ml distilled water and dilute to 1.0 liter.

c. Sodium hydroxide: Dissolve 450 g NaOH in distilled water; cool, and dilute to 1.0 liter.

d. Phosphate standard: Dissolve 1.4330 NaH_2PO_4 (dried at 105 C) in distilled water and dilute to 1.0 liter (1.0 ml = 1.0 mg PO_4).

e. Working standard: Pipet 50.0 ml of the phosphate standard into a 1.0 liter volumetric flask and dilute volume with distilled water (1.0 ml = 0.05 mg PO_4).

f. Chemicals

(1) Concentrated acids - hydrochloric, nitric and sulfuric

(2) Potassium persulfate

(3) Sodium nitrate

3. Procedure

The solid sample may be prepared for analysis either by acid digestion (a) or by dry ashing (b). Liquids are prepared by method (c). The completion of the analysis is substantially the same for all three procedures.

a. Acid digestion of solid samples: Weigh accurately 1-2 g of homogeneously ground sample (predried at 105 C) and place in a kjeldahl flask. Add 5 ml concentrated HNO_3 and 25 ml concentrated H_2SO_4 and heat until the solution boils. Then add 1 g $NaNO_3$ to the solution

and digest for two hours. After the sample is digested and cooled, add 150 ml distilled water and boil the solution for five minutes. Cool the sample and transfer to a 200 ml volumetric flask; fill to volume with distilled water and filter.

Pipet 25 ml or an aliquot of the sample (1-25 ml depending on the phosphate concentration in the sample) into a calibrated 50 ml container. Add sodium hydroxide (450 g/l), 1 ml per 5 ml of sample, to adjust the pH and dilute the sample to approximately 40 ml with distilled water. Then mix the sample, add 5 ml of vanadate-molybdate reagent, adjust the sample to the 50 ml mark with distilled water, and mix again. Allow solution to stand 15 minutes for maximum color development and read the absorbance on a spectrophotometer at 400 mu.

Calculations: Plot a standard curve from 1-25 mg/l PO_4 vs absorbance. The sample results are read directly from the standard curve taking into account the proper aliquot used for each sample.

$$\text{mg/l } PO_4 = \frac{\text{mg/l } PO_4 \times 50}{\text{Aliquot used}} \qquad \text{Percent } PO_4 = \frac{\text{mg/l } PO_4 \times 100}{\text{Wt. of sample} \times 5}$$

b. <u>Ashing of solid samples</u>: Weigh accurately 1-2 of homogeneously ground sample (predried at 105 C) and ash in a muffle furnace at 600 C for 2 hours. Cool the ash, add 40 ml HCl (1:3) and 3 drop concentrated HNO_3, and bring the solution to a boil. Cool the solution, transfer it to a 200 ml volumetric flask, dilute to volume

with distilled water and filter.

Pipet 25.0 ml or an aliquot of sample (depending on phosphate concentration in samples) into a calibrated 50 ml container and dilute to about 40 ml with distilled water. Continue the procedure as described in procedure (a), except the pH does not need adjusting in this procedure.

c. Digestion of liquid samples: Place 50 ml of the sample (or an aliquot diluted to 50 ml) into an erlenmeyer flask. Add one ml of H_2SO_4 (310 ml/l) and 0.5 g potassium persulfate, and digest the sample for 30 minutes on a hot plate. Then dilute the sample to about 20-25 ml with distilled water, filter it into a calibrated 50 ml container and dilute to approximately 40 ml with distilled, washing thru the filter paper in the process. Add 0.5 ml of NaOH (450 g/l), mix well, and continue the procedure as described in procedure (a) with the addition of the vanadate reagent.

DISCUSSION

The vanadomolybdophosphoric acid method can be used to determine phosphates in many types of solid waste. Results obtained from the methods were compared to results from two other common phosphate methods [2, 3], using standards and solid wastes samples (Table 1). The analyses indicate that the vanadomolybdophosphoric acid method compared

favorably to the other methods. This method is often more suited for solid waste samples. The concentrations of phosphates in solid waste are much higher than can be measured by the other methods without large dilution factors. The vanadomolybdophophoric acid method has a much higher range than the other methods; therefore little, if any, dilution is needed.

This method was evaluated with standards and standard addition to solid waste samples (Table 2). The results showed that at least 95 percent of the total phosphates present were recovered from all samples and standards.

TABLE 1 --- METHOD COMPARISON

Sample	Molybdate Blue Method		Ascorbic Method		Vanadate Yellow Method	
	Dil.Factor	Mg/l	Dil.Factor	Mg/l	Dil.Factor	Mg/l
Sodium Glycerophosphate (Ashed) Mg/l PO_4 in Std.						
#1 151	50	148	50	160	10	153
#2 151	50	146	50	150	10	153
#3 151	50	146	50	155	10	154
Johnson City Compost and sludge (Ashed)						
#1	10	13.8			5	18.2
#2	10	21.9			5	28.9
Johnson City Compost(70 day) (Ashed)						
#1	25	43.0	25	48.8	5	52.5
#2	25	38.0	25	43.3	5	46.0
#3	25	35.3	25	40.3	5	42.0
#4	25	39.4	25	43.3	5	46.0
#5	25	43.8	25	49.8	5	47.5
Johnson City Compost(70 day) (Digested)						
#1	50	52.5	50	46.0	5	48.5
#2	50	48.0	50	43.5	5	44.0
#3	50	47.5	50	44.0	5	46.3
Incin. Res. N.Y. #203 (Ashed)						
#1	250	228	250	217	10	220
#2	250	226	250	230	10	222
#3	250	226	250	225	10	222
Incin. Res. N.Y. #203 (Digested)						
#1	250	122	250	117	5	114
#2	250	121	250	111	5	112
#3	250	120	250	115	5	110
Food Wastes N.Y. #208 (Ashed)						
#1	100	136	100	144	10	143
#2	100	156	100	173	10	174
#3	100	130	100	151	10	155
#4	100	135	100	161	10	165
#5	100	143	100	153	10	162
#6	100	134	100	160	10	160
Quench Water Wash., D.C.	2	2.40			1	2.44

TABLE 2

PRECISION AND ACCURACY OF THE VANADOMOLYBDOPHOSPHORIC ACID METHOD

Sample	PO_4 mg/l	%PO_4	Standard Addition: PO_4 Std. Added mg/l	Standard Addition: PO_4 Std. Recovered mg/l
J. C. * compost and sludge (Ashed)				
#1	18.3	0.36	5.0	4.5
"	18.5	0.37	10.0	10.2
"	18.0	0.35	15.0	15.0
J. C. compost and sludge (Ashed)				
#2	29.0	0.41	5.0	4.9
"	28.8	0.40	10.0	9.7
"	28.8	0.40	15.0	15.1
J. C. compost(70-day) (Ashed)			Sample #2 Used-------	
#1	52.5	0.47	5.0	4.8
#2	46.0	0.46	5.0	4.8
#3	42.0	0.43	5.0	4.8
#4	46.0	0.46	10.0	9.6
#5	47.5	0.47	10.0	9.7
			10.0	9.6
J. C. compost(70-day) (Digested)			Sample #2 Used-------	
#1	48.5	0.47	10.0	9.7
#2	44.0	0.44	10.0	9.7
#3	46.3	0.46	10.0	9.7
			20.0	19.9
			20.0	19.5
			20.0	19.4
Quench Water Wash., D.C.(1967)	2.43		10.0	9.44
"	2.44		10.0	9.36
"	2.44		10.0	9.50

PRECISION AND ACCURACY OF VANADOMOLYBDOPHOSPHORIC ACID METHOD (Con'd)

Sample	PO_4 mg/l	%PO_4	Standard Addition PO_4 Std. Added mg/l	Standard Addition PO_4 Std. Recovered mg/l
N.Y. #203 Incinerator (Ashed) Residue			Sample #1 Only	
#1	222	2.22	10.0	9.4
#2	222	2.21	10.0	9.4
#3	222	2.21	10.0	9.7
			20.0	19.6
			20.0	19.6
			20.0	19.2
N.Y. #203 (Digested) Incinerator Ash			N.Y. #203 (#3 Used) 208	
#1	114	2.1	10.0	9.6
#2	112	2.20	10.0	9.9
#3	110	2.19	10.0	9.7
			20.0	19.3
			20.0	19.3
			20.0	19.5
N.Y. #208 (Ashed) Food Refuse			N.Y. #208 (#1 Used) 203	
#1	165	1.65	10.0	9.9
#2	161	1.61	10.0	9.8
#3	161	1.62	10.0	9.7
			20.0	19.2
			20.0	19.7
			20.0	19.7

*Johnson City

REFERENCES

1. Jackson, M. L. Soil chemical analysis. Prentice-Hall, Inc., 1960. p. 151-154.

2. Gales, M. E. Jr., Julian, E. C., Kroner, R. C., "A Method for the Quantitative Determination of Total Phosphorus in Filtered and Unfiltered Samples" JAWWA Vol. 58 #10 Oct., 1966.

3. "FWPCA Methods for Chemical Analysis of Water and Wastes" FWPCA Division of Water Quality Research, Analytical Quality Control Laboratory, Nov. 1969.

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